Model-free whole-powder-pattern fitting for quantitative phase analysis: a practical approach to the direct derivation (DD) method

Hideo Toraya

Rigaku Corporation, Matsubara, Akishima, Tokyo 196-8666, Japan

E-mail: toraya@rigaku.co.jp

The direct derivation (DD) method [1 – 4] for quantitative phase analysis (QPA) is based on a principle that the weight proportion of the kth component in a K-component mixture is given by S_k/a_k^{-1}, where S_k is the total sum of observed scattered intensities and a_k^{-1} is the total scattered intensity per unit weight. The magnitude of the a_k^{-1} for each component can be calculated from the numbers of electrons belonging to the atoms in the chemical formula and the chemical formula weight of that material. Therefore, weight fractions of individual components can be derived under the normalization condition if relative magnitudes of individual S_k values are obtained. Whole-powder-pattern fitting (WPPF) is a powerful technique for separating the observed diffraction pattern of the target mixture into individual component patterns, and therefore, obtaining relative S_k values. Calculated powder patterns are used for WPPF in traditional techniques such as the Rietveld or Pawley methods, while the background (BG)-subtracted observed powder patterns of single-phase component materials are used in the full-pattern-fitting method [5]. In the present procedure, the single-phase observed patterns are used for WPPF without subtracting their BG intensities. The relative magnitudes of S_k values are not affected even when the BG intensities are included if the chemical compositions of individual components are the same or similar to each other. Then we are free from the problems such as the uncertainty associated with the determination of BG height, the interaction between the BG function and the calculated/observed profile models *etc*. The present procedure gives accurate QPA results, and it is particularly advantageous when the target mixture sample contains low symmetry materials with many weak peaks, low crystalline materials, amorphous materials, and materials with no crystallographic information. Theoretical basis and experimental verification of the present procedure will be given in this report.

References

1) Toraya, H. (2016). *J. Appl. Cryst.*, **49**, 1508 – 1516.

2) Toraya, H. (2017). *J. Appl. Cryst.*, **50**, 820 – 829.

3) Toraya, H. (2018). *J. Appl. Cryst.*, **51**, 446 – 455.

4) Toraya, H. & Omote, K. (2019). *J. Appl. Cryst.*, **52**, 13 – 22.

5) Smith, D. K. *et al.* (1987). *Powder Diffraction*, **2**, 73 – 77.